Analysis of a Vitamin Mineral

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1 Introduction

A vitamin mineral sample was presented to this laboratory for trace and major element determination. The sample was previously digested by microwave heating. The assay required was Zn, Fe, Cu, Mn, I2 at % levels and Pb, Cd, Hg, Al at ppm levels in the solid sample. This report gives the best wavelengths for the determination of analytes in these matrices, as well as the operating conditions and the analytical results.

2 Principle

2.1 Technique used

The elemental analysis of solutions was undertaken by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES). The sample is nebulized then transferred to an argon plasma. It is decomposed, atomized and ionized whereby the atoms and ions are excited. We measure the intensity of the light emitted when the atoms or ions return to lower levels of energy. Each element emits light at characteristic wavelengths and these lines can be used for quantitative analysis after a calibration.

2.2 Wavelength choice

The choice of the wavelength in a given matrix can be made using the "profile" function, or by using Win-IMAGE, which is rapid semi-quantitative analysis mode using multiple wavelengths. The principle is the same in either case: record the scans of analytes at low concentration, and of the matrix. By superimposing the spectra, we see possible interferences.

3 Sample preparation

The sample was dissolved with a microwave digester (Prolabo): 1.0461 g was dissolved in 15 % HCl using a microwave vessel. A power of 50 W was applied for 10 minutes.

4 Instrument specification

The work was done on a JY 124. The specifications of this instrument are listed below in Table 1 and 2.

Table 1: Specification of spectrometer

Parameters	Specifications
Mounting	Czerny Turner
Focal length	0.64m
Nitrogen purge	Yes
Variable resolution	Yes
Grating number of grooves	2400 gr/mm

Table 2: Specification of RF Generator

Parameters	Specifications
Type of generator	Solid state
Observation	Radial
Frequency	40.68 MHz
Control of gas flowrate	Manual
Control of pump flow	Manual

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5 Operating conditions

The operating conditions are listed in Table 3 below.

Table 3: Operating conditions

Parameter	Condition
RF Generator power	1000 W
Plasma gas flowrate	12 L/min
Auxiliary gas flowrate	0 L/min
Sheath gas flowrate	0.2 L/min
Nebulizer flowrate	3.2 bars (48 psi)
Sample uptake	1 mL/min
Type of nebulizer	Concentric
Type of spray chamber	Cyclonic
Argon humidifier	No
Injector tube diameter	3.0 mm

6 Wavelength selection and analytical conditions

The analysis mode 2 is based on a gaussian curve calculation (with 7 measured points and 5 calculated points). The analysis mode 5 is a direct peaking measurement.

The most sensitive Al line at 167.020 nm was not used because of an Fe interference. The concentration range of Al in the sample allows the use of a less sensitive line.

Table 4: Analytical conditions

Element	Wavelength (nm)	PM High Voltage (V)	Slits (µm)	Analysis mode	Integration time (sec)
Al	396.152	652	20/15	2	1
Cd	228.802	703	20/15	5	6
Cu	324.754	631	20/15	2	1
Fe	259.940	541	20/80	5	4
Hg	184.890	953	20/15	5	6
I	178.218	953	20/15	2	1
Mn	259.373	511	20/80	2	1
Pb	220.353	703	20/15	5	6
Zn	202.548	591	20/80	5	4

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7 Discussion

7.1 Calibration

Standard solutions were prepared in deionized water (no digestion blank was provided). Background corrections were applied for all elements.

The calibration was made using the following standard solutions:

Table 5: Standard solutions

Element	Background Correction	Unit	Standard solutions		
			1	2	3
Al	-0.0566/0.0531	mg/L	0	3	30
Cd	0.0611	mg/L	0	0.1	1
Cu	-0.007	mg/L	0	5	20
Fe	0.0804	mg/L	0	32	160
Hg	-0.0266/0.0295	mg/L	0	0.1	1
I	0.0473	mg/L	0	0.5	1
Mn	0.0766	mg/L	0	10	60
Pb	-0.0408	mg/L	0	0.5	5
Zn	0.0851	mg/L	0	26	130

7.2 Results

Before analysis of the sample, it was diluted 10 times. The results of the analysis are presented both as the concentration in the liquid sample and in the solid sample.

Table 6: Results

Element	Result in mg/l (digested solution)	Corresponding concentration in solid sample	
Al	28.76 + 0.33	2200 + 25	mg/kg
Cd	0.095 + 0.015	7.24 + 1.17	mg/kg
Cu	218.37 + 11.77	1.67 + 0.09	%
Fe	2410 + 78	18.43 + 0.60	%
Hg	< DL	< DL	mg/kg
I	12.42 + 2.62	0.095 + 0.02	%
Mn	800 + 48	6.12 + 0.37	%
Pb	4.5 + 0.82	345 + 23	mg/kg
Zn	1753 + 308	13.41 + 2.36	%

The mercury profiles suggest the presence of this element in the sample, but the concentration is not significant to be quantified. The possibility of Hg loss during the digestion process or even during storage must be considered.

A rapid test was made in order to make the analysis of low levels of mercury possible. It is based on mercury vapor generation. For this purpose, we used the HORIBA Scientific patented CMA ("Concomitant Mercury Analyzer") accessory. This accessory allows the analysis of hydride forming elements and of the "regular" elements at the same time.(Refer to application Note 13 for more details on CMA)

8 Summary

The analysis was found to be simple with the low cost HORIBA Scientific sequential ICP spectrometer, even though both major and traces elements were required. Results were good even for elements like I_2 and Hg, which have their best wavelengths in the far UV region.

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